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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 5 : C08K 3/00, C08F 2/46

A1

(11) International Publication Number: WO 94/02540

(43) International Publication Date: 3 February 1994 (03.02.94)

(21) International Application Number: PCT/US92/06116

(22) International Filing Date: 22 July 1992 (22.07.92)

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(81) Designated States: AU, CA, JP, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LU, MC, NL, SE).

Published
With international search report.

(54) Title: HIGHLY FILLED POLYMERIC COMPOSITIONS

(57) Abstract

A highly filled composition is described containing a polymeric material, at least one filler, and at least one surface active agent. The filler(s) and surface active agent(s) are selected based upon the relationship between their pH's allowing for enhanced filler loading of the compositions. The compositions of the present invention can be used to coat electrical and electronic components or as covering substrates.

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HIGHLY FILLED POLYMERIC COMPOSITIONS

Field of the Invention

This invention relates to a highly filled coating composition. The composition is suitable for use in providing a protective barrier coating on electrical and electronic components and as an ink for printing on various substrates. The composition may include thermoset or thermoplastic polymers.

Background of the Invention

Electrical and electronic components, e.g., thick and thin film hybrid circuits, tantalum capacitors, axial leaded film-foil capacitors, ceramic disk and tubular capacitors, electrolytic capacitors, radial film foil capacitors, resistors, stacked film capacitors, and related electrical and electronic components must be, in general, provided with a protective coating. Such a coating must provide ease in handling and be capable of providing the electrical and electronic component certain desired physical, thermal, electrical insulation and moisture resistance properties. The variety of different shapes of electrical and electronic components that

are provided with a protective coating present certain unique problems. The composition used must not only provide the desired properties mentioned as a protective coating, it must also be one that can be applied to the component involved using the most suitable coating procedure. Moreover, as with any coating composition, it is desired that the protective coating be obtained in the most economical manner without compromise to the various properties desired.

In general, the protective barrier coating on an electrical or electronic component is provided by first coating the electrical and electronic component, or a portion thereof, with a resinous composition, and then heating the coated component at a suitable temperature and for a suitable length of time whereby the coating composition is cured and adheres to the underlying electrical or electronic component. The post curing step of the coating operation may be shortened in some cases by preheating the component before application of the resinous coating composition. The coating composition used may be, in some cases, either a solid, powdery composition, or, in other cases, one that is liquid, depending somewhat upon the substrate being coated. The use of such a coating composition and procedure is, however, attendant with certain disadvantages. In particular, where the resinous composition is solvent based, this may create environmental considerations. Moreover, such compositions sometimes result in the creation of

bubbles and pin-holes in the coating layer created by entrapped air during the curing cycle. Furthermore, conventional thermal curing of resinous coating compositions is not only time consuming but also costly in terms of energy consumption, space, equipment, coating material usage and personnel.

Printing and screening inks are applied to a wide variety of substrates, e.g., metals, metal alloys, paper, thermoplastic and thermosetting resin layers, etc. Nevertheless, particular problems are presented when the substrate is heat sensitive. The ink composition applied must be capable of being cured under conditions not damaging to the substrate.

The ultraviolet curing of coating compositions has been known now for sometime. Exemplary of the prior art are United States Patents Nos. 3,896,014; 4,349,605; and 4,390,401. U.S. 3,896,014 discloses liquid nail lacquer compositions which comprise as the essential components a polyene, a polythiol, a photocuring rate accelerator and, as disclosed by the patentee, a surfactant from a particular class. Among the preferred surfactants are sorbitan sesquioleate, sorbitan dioleate, sorbitan trioleate, pentaerythritol dioleate and pentaerythritol trioleate. Other surfactants found operative in the composition disclosed, but deemed by the patentee to be somewhat less efficient, include alkenyldimethylethyl ammonium bromide; di"coco"dimethyl ammonium chloride' quaternary imidazolinium salt (from stearic acid); glyceryl

monooleate; glyceryl dioleate; glyceryl trioleate and polyglyceril ester of oleic acid. Nevertheless, a host of other surfactants were discovered to be inoperative in the invention due to the fact that they were either insoluble in the composition, or they did not improve the wettability of the composition as it tended to "bead" when applied to the surface of the nail.

In U.S. 4,349,605, there are disclosed radiation curable polymeric compositions having flame retardant properties which comprise copolymers of ethylene and a comonomer which may be vinyl ester or an acrylate or a methacrylate, a hydrated inorganic filler, an alkoxy silane, and a lubricant comprising lauric acid and ethylene-bis-stearamide. The filler can, if desired, be silane treated, rather than adding the filler and silane separately to the composition. Such polymeric compositions are disclosed to be preferably cured by radiation means, although cross-linking of the polymers can also be achieved by chemical crosslinking or thermal crosslinking. According to the patentees, such polymeric compositions will hold very large amounts of filler and still provide high flexibility and a high degree of crosslinking. This is deemed by the patentees to be quite surprising as high flexibility and high crosslinking are generally incompatible, as are high crosslinking and high filler loading (which implies low crosslinkable polymer content). In compositions used for coating, e.g., extruding onto electrical wire and cables, best results, according to the

patentees, are obtained when from 44 to 80% by weight of filler in the composition, or 22 to 59% volume of filler, preferably 50 to 57% by weight of filler in the composition, or 26 to 32% volume of filler are employed.

U.S. 4,390,401 discloses ultraviolet curable coating compositions which comprise polyunsaturated polyacrylates or methacrylates and as a wetting agent and adhestive promoter an acrylate or methacrylate of a polyalkylene oxide derivative of a mono-hydric alkyl/aryl phenol.

Others in addition to the patentees in U.S. 4,349,605 have disclosed using a pretreated filler in a polymeric composition. Thus, in U.S. 2,952,595, it is disclosed that vinyltriethoxysilane treated filler, e.g., hydrated silica, in amounts from about 11 to 34% by weight of the composition or 10 to 30% volume of filler, enhances the beneficial effect that radiation treatment has on the filled polyethylene. Thus, with such treated fillers the impact strength of the compositions was improved, and the brittle point was lowered, in addition to improved torsional hepteresis.

In Japanese 56147846 (Matsushita Electric Works) there is disclosed a photocurable polyester resin composition for the production of thick sheets which comprises unsaturated polyester resin, a photopolymerization initiator, and an inorganic filler, e.g., calcium carbonate, calcium silicate,

titanium oxide, aluminum oxide, talc, clay, alumina, calcium hydroxide, and magnesium carbonate, coated with a surfactant. Those surfactants specifically disclosed are stearic acid, lauric acid, rosin, lignin, and cationic surfactant. According to the abstract, the treated filler is used at 16.6 to 80% by weight of the composition. The surface-treated filler is claimed to permit UV to penetrate the composition to deep inside, allowing production of thick sheets.

For some purposes, it would be desirable to obtain a composition which is sufficiently fluid to allow coating on a substrate yet has a volume % of filler which is sufficiently high to impart durability into the final formed coating. It is sometimes difficult to achieve the desired volume % of filler in a particular composition.

Summary of the Invention

The present invention provides a highly, filled composition having a high volume % of filler allowing for the formation of a final durable coating. The highly, filled compositions include a polymeric material at least one filler, and at least one surface active agent. The present invention is based, at least in part, on the discovery that a particular pH relationship between the filler(s) and surface active agent(s) allows for high volume % filler loading of the composition. One of the surface active agent(s) or filler(s) has an overall

pH greater than or equal to seven while the other of the surface active agent(s) or filler(s) has an overall pH of less than or equal to seven. The pH's of both components cannot be equal to 7.

The present invention also pertains to methods of making the highly, filled compositions, methods of using the highly, filled compositions, and products coated with the highly, filled compositions. The compositions can provide a protective coating on a localized area of, or can fully encapsulate, an electrical or electronic component. The compositions further can be used as a printing ink for the imprinting of various substrates.

Detailed Description

The highly filled compositions of the present invention include a polymeric material, at least one filler, and at least one surface active agent. One of the surface active agent(s) and filler(s) has an overall pH greater than seven and the other of the surface active agent(s) and filler(s) has an overall pH of less than seven. The pH of both components cannot be equal to 7. The pH relationship between the filler(s) and surface active agent(s) enhances the dispersion of the filler(s) in the composition allowing for high filler loading of the composition.

The term polymeric material for purposes of this invention includes polymers, prepolymers and combinations thereof. Prepolymers is intended to

include materials or subunits capable of being formed or polymerized into polymers, e.g., monomers, dimers, or oligomers. The polymers of the present invention further may be thermoplastic or thermoset polymers.

Thermoset Polymer Systems

Thermoset polymers are art-recognized and are extensively crosslinked polymers which do not significantly soften upon heating. Typically thermoset polymers are cured causing crosslinking forming a final coated product. The curing may be any type of curing which causes crosslinking including radiation, e.g., electron beam, catalyst, humidity or air curing. Thermoset polymer systems may include a polymer and/or prepolymer. The systems also may include additional agents which enhance the crosslinking process. Examples of thermoset polymers include the following:

phenol-formaldehyde
melamine-formaldehyde
urea-formaldehyde
polyurethane
unsaturated polyester
epoxy
phenolic anilin
furan
polyester
polyurethane
polyphenylene sulfide
polyimide

silicone
poly-p-phenylene benzobisthiazole
polyacrylate
polymethacrylate
novolac
phenolic
alkyd

The prepolymer is a material or subunit capable of being polymerized into a polymer. The term prepolymer includes monomers. For example, the prepolymer can be any mono- or multifunctional resinous materials having at least one vinyl group. Illustrative of suitable materials for use as the prepolymer are:

aliphatic urethane acrylate and methacrylate
aromatic urethane acrylate and methacrylate
polyester urethane acrylate and methacrylate
hydrocarbon urethane acrylate and methacrylate
cycloaliphatic epoxy acrylate and methacrylate
aliphatic epoxy acrylate and methacrylate
aromatic epoxy acrylate and methacrylate
amine modified epoxy acrylate and methacrylate
poly(acryloxypropylmethyl)-siloxane

poly(dimethylsiloxane)-
diphenylsiloxane) -
(methylvinyl siloxane)
polyvinylmethylsiloxane
tetramethlydisiloxane-ethylene
copolymer vinyl
terminated polydumethylsiloxane-
vinylmethyl copolymers
acrylated and methacrylated epoxy
linseed oil
vinyl unsaturated polyesters
polybutadiene acrylate and
methacrylate
polystyrene acrylate and
methacrylate
polyester acrylate and methacrylate
vinyl caprolactam
caprolactam acrylate and
methacrylate
silicone acrylates and
methacrylates
melamine acrylate and methacrylate
copolymerized amine acrylate and
methacrylate
cellulose acrylate and methacrylate
cellulose ester ether acrylate and
methacrylate
hydrocarbon acrylate and
methacrylate
polyoxyalkylated bisphenol A
acylate and methacrylate

acrylamidomethyl substituted cellulose ester acrylate and methacrylate
1-norbornene spiroorthocarbonate
2-bismethylene spiroorthocarbonate
diallyl isophthalate
diallyl maleate
diallyl phthalate

Of the above prepolymers, aromatic urethane acrylate and methacrylate, hydrocarbon urethane acrylate and methacrylate, aromatic epoxy acrylate and methacrylate, and diallyl isophthalate are preferred.

The prepolymers may be added to the composition in amounts of from 0 to 70% by weight of the composition, and preferably from 3 to 20% by weight of a coating composition. Nevertheless, as will be the case with all the components involved, the amount used will be determined somewhat by the properties desired in the cured composition, which, in turn, will be determined by the particular application involved, i.e., whether the composition is an ink composition or a composition to be used for coating electronic or electrical components. If the latter, the particular formulation desired will also be influenced by the particular components to be coated, including the shape thereof, and the manner of coating to be employed, e.g. dip coating, end pour, roll coating, etc.

The prepolymer may be a monomer. Monomers used in the compositions of the invention can be mono- or multifunctional, e.g., mono- or multi-functional vinyl monomers. Preferably, the monomer has at least one vinyl, or unsaturated, group whereby it will, on curing as later more specifically disclosed, cross-link and form a coating or ink, e.g., a thermoset coating or ink. Monomers that will be found suitable in the practice of the invention are:

cyclohexyl acrylate
ethyl acrylate
butyl acrylate
2-ethylhexyl acrylate
2-hydroxyethyl acrylate
hydroxypropyl acrylate
N-vinyl-2-pyrrolidone
2-ethoxy ethoxyethyl acrylate
isobornyl acrylate
methyl acrylate
dimethylaminoethyl acrylate
diethylaminoethyl acrylate
tetrahydrofurfuryl acrylate
n-hexyl acrylate
n-lauryl acrylate
2-phenoxyethyl acrylate
furfuryl-2-acrylate
3-dimethylamino neopentyl acrylate
2-cyanoethyl acrylate
benzyl acrylate
2-N-morpholinoethyl acrylate

2-tert-butyl aminoethyl acrylate
N-benzyl methacrylamide
2-(1-aziridinyl)-ethylacrylate
methacrylopyethyl phosphate
bismethacrylopyethyl phosphate
N-(iso-butoxymethyl) acrylamide
3-phenoxy-2-hydroxypropyl acrylate
3-methacryloxypropyltris
(trimethylsiloxy) silane
3-methacryloxypropylpentamethyl-
disiloxane
calcium methacrylate
chromium (III) dimethacrylate
sodium methacrylate
magnesium methacrylate
silicone (IV) trimethacrylate
tributyl tin methacrylate
zinc methacrylate
zirconium (IV) dimethacrylate
2-methacryloxyethyl phenylurethane
dicyclopentenyl acrylate
monomethoxy tripropylene glycol
monoacrylate
monomethoxy neopentyl glycol
ethoxylate monacrylate
monomethoxy 1,6 hexanediol
monoacrylate
propargyl methacrylate
isocyanotoethyl methacrylate
2-N-morpholinoethyl acrylate
allyl acrylate
styrene

vinyl acetate
N-cyclohexyl methacrylamide
N, N'-isovalerylidene-
bis-methacrylamide
3,3,5 trimethylcyclohexyl acrylate
dihydrodicyclopentadienyl acrylate
maleic acid-mono-
2-acryloxyethylester
maleic acid-mono-2-
methacryloxyethylester
n-octyl acrylate
diallyl fumarate
tripropylene glycol diacrylate
1,6 hexanediol diacrylate
N, N'-methylenebisacryamide
triethylene glycol diacrylate
ethylene glycol diacrylate
polyethylene glycol diacrylate
1,3 butylene glycol diacrylate
1,4 butanediol diacrylate
diethylene glycol diacrylate
diphenylvinylchlorosilane
1,3 divinyl-1,3 diphenyl-1,3
dimethyldisiloxane
1,3-divinyltetramethyldisiloxane
neopentyl glycol diacrylate
ethoxylated bisphenol A diacrylate
caprolactone diacrylate
2-methyl-2-carboxy propanediol
diacrylate
thiodiglycol diacrylate
allyl acrylate

dianol diacrylate
3-methylbutene-2-yl-1-acrylate
N,N' hexamethylene-
bis-methacrylamide
monomethoxy trimethylolpropane
diacrylate
monomethoxy pentaerythritol
propoxylate triacrylate
monomethoxy glycerol propoxylate
diacrylate
trimethylol propane triacrylate
pentaerythritol triacrylate
trimethylolpropane ethoxylate
triacrylate
polyether triacrylate
mono-methoxy pentaerythritol
propoxylate triacrylate
triallylcyanurate
melamine acrylate
tris (2-hydroxy ethyl)
isocyanurate trimethacrylate
1,6 hexanediol diglycidyl ether
diacrylate
triallyl trimellitate
pentaerythritol tetraacrylate
dipentaerythritol monohydroxypenta
acrylate
dipentaerythritol pentaacrylate
dipentaerythritol hexaacrylate
vinyl tris (isopropenoxy) silane
1,3,5 trivinyl-1,1,3,5,5-
pentamethyltrisiloxane

The preferred monomers are alpha, beta unsaturated carboxy compounds. Most preferred monomers are acrylates, and alkacrylates such as isobornyl acrylate, dicyclopentenyl acrylate, 1,6-hexanediol diacrylate, and trimethylol propane triacrylate and alkacrylate relatives of all of the foregoing.

In general, these vinyl unsaturated monomers may be added to the composition in amounts ranging from 1 to 70% by weight of the composition, and preferably from about 3 to about 20% by weight of a coating composition used.

Photoinitiators and sensitizers suitable for use in the radiation curable compositions of this invention are any of those commonly used for this purpose. Thus, compositions of this invention can include various known photosynergists, e.g. alkylamines, alkanolamines, and morpholines, or commonly used photoinitiators, e.g. either those involving hydrogen abstraction or intramolecular photocleavage. Examples of suitable photosynergists and sensitizers are:

alkylamines
alkanolamines
morpholines
2-(n-butoxy) ethyl
4-dimethylaminobenzoate
ethyl para dimethyl amino benzoate

2-(dimethylamino) ethyl benzoate
diethanol amine
monoethanol amine
dimethylethanol amine
dimethylaminoethyl acrylate and methacrylate
diethylaminoethyl acrylate and methacrylate
N-methyldiethanolamine
2-ethylhexyl p-(N, N-dimethylamino) benzoate
2,2 dithiobisbenzothiazole
bis-ethylamino benzophenone

Examples of photoinitiators which can be used in the practice of the invention include:

N,N'dimethylaminobenzophenone
d,l-camphorquinone
2-isopropylthioxanthone
4-isopropylthioxanthone
benzophenone
benzil
2-chlorothioxanthone
2-methylthioanthone
chlorine functional substituted benzophenone
halogen substituted alkyl-arylketone
4-benzoyl-4-methyl-diphenyl sulphide
2,4,6 trimethylbenzoyldiphenylphosphine oxide
methyl phenylglyoxlate
dibenzosuberone

isobutyl benzoin ether
isopropyl benzoin ether
benzoin ethyl ether
benzoin methyl ether
1-phenyl-1,2
   propane-dione-2-(0-ethoxycarbonyl)
   oxime
2-2, dimethoxy-2, phenyl-acetophenone
1,hydroxy cyclo hexyl phenyl ketone
2,2 diethoxyacetophenone
2-hydroxy-2 methyl-1 phenyl propan-1-one
2-methyl-1-(4-methylthio) phenyl)-2-(4
   morpholinyl)-1-propanone
4,4'bis-(dimethylamino) benzophenone
dibenzosuberone
valerophenone
alpha-tetralone
9-fluorenone
tri-o-tolylphosphine
benz [a] anthacene-7,12-dione
7-H-benz [de] anthracene-7-one
hexanophenone
4,4'dimethoxybenzophenone
4-methoxybenaldehyde
2,4 dimethylthioxanthone
2,3 butanedione
alpha, alpha, alpha
   trichloroacetophenone
dimethylsilane-dimethylsiloxane
   copolymer

The preferred photoinitiators for use in the compositions are 2-2 dimethoxy-2, phenyl-acetophenone, 2 hydroxy-2 methyl-1 phenyl propan-1-one, 2 ethylhexyl p-(N, N-dimethylamino) benzoate and 2 isopropylthioxanthone.

In general, the amount photoinitiator or sensitizer used will depend somewhat upon that particular one chosen, and the formulation of the compositions, e.g., monomers and prepolymers involved. However, as a rule of thumb the photoinitiator used will depend upon the desired depth of cure, reaction kinetics, solubility in the system, shelf life of the composition and wavelength of radiation used. The photoinitiators may be added to the composition in amounts ranging from 1 to 15% by weight of the composition; and preferably from about 2 to about 6% by weight of a coating composition.

It will be readily appreciated by those skilled in the art that such photoinitiators and sensitizers as set forth above will not be incorporated in any composition where such is to be cured by electron beam radiation.

Although not a critical feature of the invention, the addition of peroxides or other known accelerators to the compositions disclosed herein will hasten their cure. Thus, in the case where the thickness of the coating layer is to be greater than about 10 mils, it may be desirable to include in the composition such an accelerator. Examples of

peroxides that will be found suitable for this purpose, and as an optional component, are:

diacyl peroxide
ketone peroxides
peroxydicarbonates
peroxyesters
dialkyl peroxides
hydroperoxides
sulfonyl peroxides
peroxyketals
symmetric and unsymmetric azonitrile
dialkyl peroxydicarbonate
tetra-alkyl peroxyester
tetra-alkyl hydroperoxide
peroxyacids

Thermoplastic Polymer Systems

Thermoplastic polymers are art-recognized and are polymers which soften upon heating and harden upon cooling. The softening/hardening cycles may be repeated many times. Examples of thermoplastic polymers include the following:

polyethylene
polypropylene
polystyrene
polyvinyl chloride
polyvinyl alcohol
polytetrafluoroethylene
polytetrafluoroethylene-co-ethylene
polymethyl methacrylate

polymethyl methacrylate-co-acrylonitrile
polystyrene
polystyrene/polybutadiene
polystyrene/polybutadiene-co-
arcylonitrile
polybutadiene
polystyrene-co-acrylonitrile
polyoxymethylene
polyethylene terephthalate
polycarbonate
poly e-caprolactam
polyhexamethylene adipamide
polysulfone
cellulose acetate
cellulose acetobutyrate
cellulose
polyisoprene
polybutadiene-co-styrene
polybutadiene-co-acrylonitrile
polychloroprene
polyisobutene-co-isoprene
bromo butyl rubber
polyethylene chlorosulfonated
polyethyl acrylate
polyethylene-co-vinyl acetate
polyethylene-co-propylene
polyurethane rubber
polysulfide rubber
silicone rubber
polyvinyl butyrate
polyvinyl fluoride
polyvinylidene fluoride

polyester
polyacetal
polyamide
nitrile
cellulose nitrate
acrylonitrile-butadiene-styrene
polysulphone
polymethylpentene
ethylene/vinyl acetate copolymer
polyoxymethylene
polyethylene oxide
polyimide
ethyl cellulose
cellulose propionate
cellulose acetate butyrate
polyvinyl acetate
styrene-butadiene copolymers
polyvinyl acetate copolymers
polymethyl methacrylate copolymers

Thermoplastic polymer systems typically include a solvent which is evaporated from the system producing a final coating. The solvent can be evaporated at ambient temperature or heating may be used to shorten the evaporation time. The solvents used in a thermoplastic system may be any solvent capable of solvating the selected polymer. One of ordinary skill in the art would be able to select a useful solvent for a particular polymer. Examples of solvents which may be used in this invention include:

polytetrafluoroethylene
neopentane

polyacrylonitrile
water
n-pentane
n-hexane
n-octane
diisopropylketone
cyclohexand
carbon tetrachloride
toluene
xylene
isopropyl alcohol
methyl ethyl ketone
ethyl acetate
dioxane
acetone
pyridine
ethanol
methanol
glycerol
dibutyl phthalate
trixylyl phosphate
butyl acetate

The following description applies to compositions containing either thermoset or thermoplastic polymers. The type of polymer selected does not effect the ability of the pH relationship between the surface active agent(s) and filler(s) to enhance the filler loading of the composition.

Surface Active Agents

The compositions disclosed herein include at least one surface active agent. Surface active agents are art-recognized components. The total amount of surface active agent(s) are preferably added to the composition in amounts ranging from about 0.5 and about 4% by weight of the composition. Such agents include various of those components that are variously referred to in the art as surfactants, dispersants or dispersing agents, emulsifiers, suspension agents and solubizers.

In some cases, the use of more than one surface active agents is preferred for best results. Silane, for example, does not, alone, provide maximum loading of fillers having a negative charge. Such fillers, e.g. calcium metasilicate (wollastonkup), disperse only to a certain loading, e.g. about 42% by weight of the composition, or 52% volume of filler, and above that loading, the filler tends to agglomerate. Nevertheless, when a different surface active agent or a second surface active agent, e.g. phosphate acid ester (such as GAFAC, RE-610), is added to the composition, the filler particles then disperse from one another, with attendant reduction in viscosity (see Table 1, Experiment No. 1).

The surface active agent to be used can, if desired, be used to coat the filler particles prior to their introduction in the composition. Such a pretreatment of fillers is known, and silane treated fillers are available commercially. Moreover, where two surface active agents are desired in the

composition, to obtain maximum loading and polymer properties, the use of a surface active agent coated filler will be found most advantageous.

Examples of surface active agents that can be used in the invention are:

fish oil (one or more of oleic, palmitic and stearic acid)
anionic polyelectrolyte
linoleic acid
oleic acid
palmitic acid
stearic acid
carboxylic acids
oleoyl acids
stearoyl sarcosine
sodium salt of polycarboxy...
anionic/nonionic surfactant blend
fatty alcohol ethoxylates
organic phosphate ester acid
acid phosphate ester of fatty alcohol alkoxylate
silanes
titanates

Examples of various surface active agent(s) that can be used in the invention to disperse fillers whose pH is greater than or equal to 7 are:

fish oil (one or more of oleic, palmitic and stearic acid)
anionic polyelectrolyte

linoleic acid
oleic acid
palmitic acid
stearic acid
carboxylic acids
oleoyl acids
stearoyl sarcosine
sodium salt of polycarboxylic acids
anionic/nonionic surfactant blends
fatty alcohol ethoxylates
organic phosphate ester acid
methacryloyloxyethane 1, 1
   diphosphoric acid
vinyl (acrylic or methacrylic)
   substituted polyaliphatic
   phosphoric acid
vinyl (acrylic or methacrylic)
   substituted aromatic
   phosphoric acid
vinyl (acrylic or methacrylic)
   substituted bisphenol A
   phosphoric acid
vinyl (acrylic or methacrylic)
   substituted polyethoxy
   phosphoric acid
vinyl (acrylic or methacrylic)
   substituted polypropoxy
   phosphoric acid
acid phosphate ester of fatty
   alcohol alkoxylate
maleic mono acryloyloxyethylester

maleic mono methacryloyloxyethylester
phthalic mono methacryloyloxyethylester
methacrylic acid

A most preferred surface active agent is the phosphate acid ester GAFAC, RE-610 to disperse fillers whose pH is greater than or equal to 7. This surface active agent whose pH is less than 7 results in good dispersion of the calcium metasilicate filler whose pH is 9.9 when introduced into the composition. Other phosphate acids esters which are useful as dispersants are GAFAC: RS-610, RS-410, RE-410, RM-710, RM 510, RM 410, RS-710, RP 710, PE-510, RD-510, RA-600, and Emphos, PS-21A, and also non-phosphate acid esters such as: fish oil, oleic acid, palmitic acid, and stearic acid. The effect each dispersant has on silane treated calcium metasilicate (400-NYAD-1024) and untreated calcium metasilicate (400-NYAD) are given in Table 1 and Table 2, respectively. Table 1 is a list of dispersants and their effect on the treated filler as illustrated by the high volume % filler. The dispersants used herein give higher volume % filler loadings than the previous mentioned dispersants in U.S. Patent Nos. 3,896,014; 4,349,605; and 4,390,401. Table 2 is a selected list of dispersants and their effect on untreated filler. The dispersants used gave a higher % volume filler than previously disclosed in the patents just listed.

Examples of surface active agents that can be used in the invention to disperse fillers whose pH is less than or equal to 7 are:

polypropoxyglycol methyl diethyl quaternary ammonium chloride
polypropoxyglycol methyl diethyl quaternary ammonium phosphate
polypropoxyglycol methyl diethyl quaternary ammonium acetate
cationic/nonionic surfactant blends
2-trimethylammonium ethylmethacrylate chloride, or phosphates, or acetates
N-trimethylammonium propylmethacrylamide chloride, or phosphates, or acetates
2-trimethylammoniumethyl acrylate chloride, or phosphate, or acetate

The most preferred surface active agent is the quaternary ammonium salt Emco, CC-42. This surface active agent whose pH is neutral or slightly basic gives good dispersion with carbon black (Monarch 1,000) whose pH is 2.5 when introduced into the composition. Also, the quaternary ammonium salt gives good dispersion in systems whose filler is a metal and slightly acidic, i.e., electrolytic copper, and nickel. Equally good results are achieved when the metal or metal oxide is neutral. Neutral metals such as copper, silver, palladium, gold, tungsten, platinum, etc. are dispersed in the quaternary ammonium salt.

Other quaternary ammonium salts which are equally useful as dispersants are Emco: CC-9, CC-55, and the low molecular weight quaternary ammonium salts of methacrylic (acrylic) functionality, such as: BM-604, 2-trimethylammonium ethylmethacrylate chloride, or phosphate, or acetates; BM-613, N-trimethylammonium propylmethacrylamide chloride, or phosphates, or acetates; and BM-607, 2-trimethylammoniumethyl acrylate chloride, or phosphates, or acetates. The effect each dispersant has on carbon black, nickel, and copper are given in Table 3.

When the fillers are of opposite pH values or one filler is either acidic or basic and the other is neutral then one of the surface active agent(s) must have a pH greater than or equal to 7 and the other surface active agent must have a pH less than or equal to 7. Table 4 illustrates the combining of fillers whose pH values are opposite or where one filler, either acidic or basic, is combined with a neutral filler (inorganic).

The monomer, photoinitiator, and surface active agent were mixed at ambient temperature for 15 minutes. Filler was added gradually over 30 minutes, whereby the mixture temperature increased to 85° due to the high shear generated during mixing. The mixture composition upon mixing with dispersant remained fluid, whereas, the mixture composition without dispersant was a solid compact cake of high viscosity.

Table 5 is a summary of materials referred to herein by trade or code names listing corresponding chemical names therefor and available sources of supply.

Table 1

Effect of Dispersant on Silane Treated Calcium Metasilicate Filler

Experiment Number

Materials	1	2	3	4	5	6	7	8	9	acid value range	average molecular weight
	%wt.	%wt.	%wt.	%wt.	%wt.	%wt.	%wt.	%wt.	%wt.		
Monomer											
HDODA	12.87	12.9	14.8	16.4	12.8	12.8	12.8	12.8	12.8		
Photoinitiator											
1173	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6		
Dispersants											
Re-610	1.6									62.72	837
Rs-610		1.6								75-85	701
Ps-21A			1.8							130	431
RS-410				3.4						95-115	534
RE-410					3.2					85-100	606
RM-710						1.6					1516

Table 1 (continued)

Materials	1 %wt.	2 %wt.	3 %wt.	4 %wt.	5 %wt.	6 %wt.	7 %wt.	8 %wt.	9 %wt.	acid value range	average molecular weight
RM-510							1.6			45-55	1122
RM-410								5.1		51-64	975
BH-650									19.4	370-390	147.6
Filler											
400-NYAD-10024	84.0	83.9	81.8	78.7	82.3	82.3	82.6	82.0	82.0		
VOLUME % FILLER	65.9	65.7	62.4	63.3	65.5	65.6	60.8	47.2			

Table 1 (continued)

Materials	10 %wt.	11 %wt.	12 %wt.	13 %wt.	14 %wt.	15 %wt.	16 %wt.	17 %wt.	18 %wt.	acid value range	average molecular weight
Monomer											
HDODA	12.8	12.8	12.8	12.8	12.8	12.8	12.8	12.8	12.8		
Photoinitiator											
1173	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6		
Dispersants											
RS-710	2.5									58-70	876
RP-710		5.0								85-100	575
PE-510			5.5							49-59	1039
RD-510				4.9						100-115	522
RA-600					7.3					100-115	522

Table 1 (continued)

Materials	10 %wt.	11 %wt.	12 %wt.	13 %wt.	14 %wt.	15 %wt.	16 %wt.	17 %wt.	18 %wt.	acid value range	average molecular weight
RK-500						11.8				132-145	405
BI-750							15.9			250-275	214
2-ethyl-hexoic acid								9.7		389	144
myristic acid									1.6	247	227
Filler 400-NYAD-10024	82.4	82	82	82	82	82	82	81	52.7		
VOLUME % FILLER	64.3	60.9	60.3	61.1	58.2	53.6	50	55.3	54.8		

Table 1 (continued)

Materials	19 %wt.	20 %wt.	21 %wt.	22 %wt.	23 %wt.	24 %wt.	acid value range	average molecular weight
Monomer								
HDODA	12.8	12.8	12.8	12.8	12.8	12.8		
Photoinitiator								
1173	1.6	1.6	1.6	1.6	1.6	1.6		
Dispersants								
palmitic acid	1.9						218	257
steric acid		1.8					198	283
oleic acid			1.6				202	278
fish oil				1.6			---	---
Z-6030					2.4		---	---
Filler								
400-NYAD-10024	77.2	78	78.6	80	72.22	42.2		
VOLUME %	63.6	63.9	64.4	64.8	49.6	51.9		

Table 2

Effect of Dispersant on Untreated Calcium Metasilicate Filler

Experiment Number

Materials	1 %wt.	2 %wt.	3 %wt.	4 %wt.	5 %wt.	6 %wt.	7 %wt.	8 %wt.	9 %wt.	10 %wt.	acid value range	average molecular weight
Monomer												
HDODA	12.8	12.8	12.8	12.8	12.8	12.8	12.8	12.8	12.8	12.8		
Photoinitiator												
1173	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6		
Dispersant												
RE-610	1.6										62-72	837
Rs-610		1.6									75-85	701
Ps-21A			1.6								130	431
RE-410				3.5							85-100	606
fish oil					1.6						---	---

Table 2 (continued)

Materials	1 %wt.	2 %wt.	3 %wt.	4 %wt.	5 %wt.	6 %wt.	7 %wt.	8 %wt.	9 %wt.	10 %wt.	acid value range	average molecular weight
RA-600						8.5					100-115	522
mysteric acid							1.6				247	227
oleic acid								1.6			202	278
Z-6030									2.6		---	---
filler 400 NYAD	83.6	82.0	80.7	81.5	78.0	81.5	51.0	77.6	48.2	41.2		
VOLUME %	65.8	65.4	65	62.7	62.3	56.8	54	64.1	51.1	51.4		

Table 3

Effect of Dispersant on Carbon, Copper and Nickel

Experiment Number

Materials	1 %wt.	2 %wt.	3 %wt.	4 %wt.	5 %wt.	6 %wt.	7 %wt.	8 %wt.	9 %wt.	acid value range	average molecular weight
<u>Monomer</u>											
HPA	8	8	8	5	5	5	5	5	5		
<u>Photoinitiator</u>											
1173	1.0	1.0	1.0	0.5	0.5	0.5	0.5	0.5	0.5		
<u>Dispersants</u>											
CC-42	1.6			0.2			0.2			N/A	2,500
CC-9		1.6			0.2			0.2		N/A	2,500
CC-55			1.7			0.2			0.2	N/A	2,500
<u>Filler</u>											
Carbon Monarch 1000	89.4	89.4	89.3								
Copper R-290				94.3	94.3	94.3					
Nickel PN200							94.3	94.3	94.3		
VOLUME % FILLER	83.4	83.4	84.7	66.5	66.5	66.5	66.5	66.5	66.5		

Table 4

Effect of Dispersant on Filler Whose pH > 7 and Filler Whose pH < 7

Materials	1 % wt.	2 % wt.
Monomer		
HPA	7.0	5.0
Photoinitiator		
1173	0.5	0.5
Dispersant pH < 7		
RE-610	0.3	0.3
Filler pH > 7		
400 NYAD	50.3	50.3
Dispersant pH > 7		
CC-42	0.5	0.1
Filler pH < 7		
Carbon Monarch 1000 (pH < 7)	41.4	
Nickel PN 200 (pH=7)		43.8
Volume % Filler	83.8	80.2

Table 5

Materials Used

Code	Trade Name/Chemical Name	Company
NYCO 400 Wallastonite	calcium metasilicate	NYCO
NYCO 400 Wollastokup 10024	calcium metasilicate (silane treated filler with Z-6030)	NYCO
231 Lupersol	(1, 1-di-t-butylperoxy) -3, 3, 5 trimethylcyclohexane	Penwalt Corp.
Z-6030	-methacryloxypropyltrimethoxysilane	Dow Corning
Anionic GAFAC[R]	Surfactant	GAF Chemicals

poly(oxy-1, 2-ethanediyl), alpha-(dinonylphenyl)-omega-hydroxy, phosphate; common names:

nonyl nonoxynol-7-phosphate	RM-410
nonyl nonoxynol-10-phosphate	RM-510
nonyl nonoxynol-15-phosphate	RM-710

poly(oxy-1, 2-ethanediyl), alpha-(nonylphenyl)-omega-hydroxy, phosphate; common names:

nonoxynol-9-phosphate	RE-610
nonoxynol-4-phosphate	RE-410

poly(oxy-1, 2-ethanediyl), alpha-tridecyl-omega-hydroxy, phosphate; common names:

trideceth-6-phosphate	RS-610
trideceth-4-phosphate	RS-410
trideceth-7-phosphate	RS-710

poly(oxy-1, 2-ethanediyl), alpha-dodecyl-omega-hydroxy, phosphate; common names:

polyoxyethylene lauryl ether phosphate RD0510

poly(oxy-1, 2-ethanediyl), alpha-phenyl-omega-hydroxy, phosphate or, polyoxyethylene phenyl ether phosphate RP 710

Table 5

Materials Used

Code	Trade Name/Chemical Name	Company
NYCO 400 Wallastonite	calcium metasilicate	NYCO
NYCO 400 Wollastokup 10024	calcium metasilicate (silane treated filler with Z-6030)	NYCO
231 Lupersol	(1, 1-di-t-butylperoxy) -3, 3, 5 trimethylcyclohexane	Penwalt Corp.
Z-6030	-methacryloxypropyltrimethoxysilane	Dow Corning
Anionic GAFAC^{R}	Surfactant	GAF Chemicals

poly(oxy-1, 2-ethanediyl), alpha-(dinonylphenyl)-omega-hydroxy, phosphate; common names:

nonyl nonoxynol-7-phosphate	RM-410
nonyl nonoxynol-10-phosphate	RM-510
nonyl nonoxynol-15-phosphate	RM-710

poly(oxy-1, 2-ethanediyl), alpha-(nonylphenyl)-omega-hydroxy, phosphate; common names:

nonoxynol-9-phosphate	RE-610
nonoxynol-4-phosphate	RE-410

poly(oxy-1, 2-ethanediyl), alpha-tridecyl-omega-hydroxy, phosphate; common names:

trideceth-6-phosphate	RS-610
trideceth-4-phosphate	RS-410
trideceth-7-phosphate	RS-710

poly(oxy-1, 2-ethanediyl), alpha-dodecyl-omega-hydroxy, phosphate; common names:

polyoxyethylene lauryl ether phosphate RD0510

poly(oxy-1, 2-ethanediyl), alpha-phenyl-omega-hydroxy, phosphate or, polyoxyethylene phenyl ether phosphate RP 710

Table 5

Materials Used

Code	Trade Name/Chemical Name	Company
3, 6, 9, 12-tetraoxyhexadecan-1-ol, 15 methyl, dihydrogenphosphate and monohydrogen phosphate; or, isoamyl alcohol, ethoxylated, phosphate-BI750		
polyphosphoric acids; 2-Butoxyethyl esters polyoxyethylene butoxyethyl ether phosphate, BH-650		
poly(oxy-1, 2-ethanediyl), alpha-hexyl-omega-hydroxy-, phosphate; polyoxyethylene hexyl ether phosphate, RK-500		
Fish oil	oleic acid, palmitic acid and steric acid	H.J. Baker & Bro., Inc.
PS-21A,	Emphos, phosphate acid ester,	Witco Chemical
1173	Darocur, 2-hydroxy-2-methyl-1-phenyl-propan-1-one	EM
1573	Epoxy acrylate Resin	Cargill, Inc.
HDODA	1, 6 hexanediol diacrylate	Interez
TMPTA	trimethylolpropane triacrylate	Interez
Fc430	Fluorad, fluorinated hydrocarbon	3M
Oncor	Thermoguard S, antimony trioximide	M&T Chemicals Inc.
Air Out	petrolium ether	Furane Products Co.
IR-651	Irgacure, 2, 2 dimethoxy-2, phenyl-acetophenone	Ciby Geigy

Table 5 (cont'd)

Materials Used

Code	Trade Name/Chemical Name	Company
DCPA	dicyclopentenyl acrylate	Alcolac
DBDPO	decabromodiphenyl oxide	Great Lakes
ITX	isopropylthioxanthone	Ward/ Blenkinship
TBP	TRIGONOX-C t-butyl peroctoate	Noury Chemical Co.
3600	Celrad, amine modified diacrylate ester of bisphenol A epoxy resin	Interez
Cab-o-sil	fumed silica	Cabot
325, NYAD	calcium metasilicate	NYCO
325 Wallastokup-10022 NYAD	calcium metasilicate	NYCO
L-722 Silwet	polydimethylsiloxane	Union Carbide
MK	4,4-Bis(dimethylamino)-benzophenone	Eastman
Black-F2302	CrCuMnMo	Ferro Color Corp.
Blue-F5203	CoCrAl	Ferro Color Corp.
Green-V7687	CrCoMgAl	Ferro Color Corp.

Table 5 (cont'd)

Materials Used

Code	Trade Name/Chemical Name	Company
EPB	ethyl-para-dimethylaminobenzoate	Ward/ Blenkinsnop
HPA	Hydroxypropyl acrylate	Rohm Tech
PETA	Pentaerythritol triacrylate	Sartomer
CC-42	Polypropoxyglycol methyl diethyl quaternary ammonium chloride	Witco
CC-55	Polypropoxyglycol methyl diethyl quaternary ammonium acetate	Witco
CC-9	Polypropoxyglycol methyl diethyl quaternary ammonium phosphate	Witco
Monarch 1000	Carbon black	Cabot
R-290	Copper powder	U.S. Bronze
PN200	Nickel powder	Ametek
BM-605	Dimethylaminoethyl acrylate	Rhom Tech
BM-604	2-trimethylammonium ethylmethacrylate chloride	Rhom Tech
BM-613	N-trimethylammonium propylmethacrylamide chloride	Rhom Tech
BM-607	2-trimethylammoniumethyl acrylate chloride	Rhom Tech

Table 5 (cont'd)

Materials Used

Code	Trade Name/Chemical Name	Company
Brown-F6114	CrFeSi	Ferro Color Corp.
Yellow-V9400	NiSbTi	Ferro Color Corp.
TiO2	titanium dioxide	DuPont
ZnS	zinc sulfide	Aceto Chemical
Lucirin LR8728	2,4,6 trimethylbenzoyldiphenyl-phosphine oxide	BASF
DMAEA	demethylaminoethyl acrylate	CPS Chemical Co.
Butvar (B-79)	Polyvinyl butyral	Monsanto
MEK	methyl ethyl ketone	Ashland
Tol	toluene	Ashland
DBP	dibutyl phthalate	Monsanto

Filler

A filler is an inorganic material (as compared to organic materials composed of carbon, oxygen, hydrogen or nitrogen) capable of occupying a percent of the volume of a composition. The amount of surface active agent used in any particular formulation will depend to some extent on the particular filler used, the process of manufacturing the filler, its surface area, etc., and the amount of filler to be loaded in the composition. The amount filler used, in turn, will depend somewhat upon the particular application for the coating composition. Where a more fluid flowing coating is desired, for example, in potting electrical and electronic components, a lesser amount of filler in general will be used.

Examples of filler that can be used in the compositions of this invention whose pH are greater than or equal to 7 are:

barium sulfate
talc
sodium carbonate
zinc oxide
silica
silicates
alumina
aluminates
beryllia
metaborates
calcium sulfate
aluminum silicate

phosphates
metasilicates
zirconates
metal oxides
lithium aluminum silicate
wollastonite
titanates

Likewise, examples of fillers that can be used in the compositions of this invention whose pH are less than or equal to 7 are:

carbon black
copper
iron oxide
palladium
silver
platinum
glass
nickel
gold
tantalum
tungsten
iron
molybdenum
cadmium
metals
metal oxides
boron
aluminum
titanium
iridium

In general, the compositions of this invention may contain a filler from about 20 to about 90% of filler by weight of the composition, preferably from about 25 to 84% by weight of the composition. Where such compositions are used for coating tantalum capacitors, ceramic disc capacitors, radial film foil capacitors, hybrid circuits, etc., less than about 59% by weight of calcium metasilicate filler in the polymeric composition results in polymer degradation with formation of cracks in the coating when such coating is subject to 125°C for 4,000 hours, a specification which must be met in this application. To some extent the amount filler in any composition will depend upon the particular application therefor, i.e., what electrical and electronic component is to be coated, and what physical properties are desired in the cured, polymerized, hardened coating. Furthermore, the manner of coating the substrate will also determine to some extent the concentration of filler. In general, where the coating composition is used to end pour an electrical and electronic component less concentration of filler will be desired than in the case of encapsulating a tantalum capacitor.

pH Relationship of Surface Active Agent and Filler

The pH relationship between the surface active agent and the filler is an important aspect of this invention. For ease of discussion, either the singular form of "filler" and "surface active agent" will be used but the form is intended to encompass a

single filler or surface active agent or a plurality of such agent. The filler and surface active agent are selected such that the overall pH of one of the component is less than or equal to 7 while the overall pH of the other components is greater than or equal to 7. The overall pH of both components cannot equal 7. The language overall pH is used to cover the situation where more than one surface active agent or filler is used in the composition. The overall pH is the combined pH of more than one surface active agent or more than one filler. The preferred pH relationship of the present invention is where one of the components has an overall pH greater than 7 while the other component has an overall pH less than or equal to 7. The most preferred pH relationship is where one of the components has a pH greater than 7 while the other component has a pH less than or equal to 7.

<u>Volume % of Filler</u>

The volume % of filler in the present invention also is an important aspect of the present invention. The volume % of filler in the composition is sufficiently high to impart durability into a coating formed from the composition. A durable coating is intended to include coatings which do not crack under high humidity conditions (70% relative) at 85°C. The volume % of filler is calculated as follows: the volume % filler is equal to the volume of the filler expressed in cubic centimeters divided by the volume of filler and residual organic material, both expressed in cubic centimeters, whereby the result is multipled by 100. The volume %

filler of the present invention may be greater than about 50%, preferably greater than about 60%, more preferably greater than about 75%, most preferably greater than about 90%. The term "about" is intended to include a reasonable range on either side of the explicitly stated numerical range, e.g., ±5%, most preferably ±3%.

Additional Components

In addition to the aforesaid essential ingredients, other components such as disclosed hereinafter can be included in the composition, as desired, and so long as they do not interfere with the curing process. Thus, for example, it may be desirable for a particular application, to include in the composition one or more of the following components: a wetting agent, a plasticizer, a leveling agent, a thixotropic agent, a flame retardant, an adhesion promoter, a stabilizer, or an inhibitor, all of which are commonly used in the formulation of coating compositions and inks, to afford certain desired physical properties thereto. To further illustrate the various other additives that may be incorporated in the composition of the invention, the following is given:

Wetting Agents: Examples of various wetting agents that can be used in the invention are: polyethylene glycol fatty esters, nonyl phenol ethylene oxide, fluorinated hydrocarbons, 2,2,3,3 tetrafluoropropylmethacrylate and acrylate, fluorinated alkyl polyoxyethylene ethanol, polypropoxy quaternary ammonium acetate,

polymethylsiloxane, and ammonium salt of sulfated nonylphenoxypoly (ethyleneoxy) ethanol. The preferred wetting agents are fluorinated hydrocarbons. Fc-430 is the fluorinated hydrocarbon used herein that is soluble in the composition and, likewise, lowers the composition's surface tension.

Plasticizers: Examples of various plasticizers that can be used in the invention are: adipates, azelates, benzoates, epoxidized linseed oil, hydrocarbons, phosphates, polyesters, phthalates, trimellitates, aliphatic siloxanes, nonionic (polyethylene oxides), anionic (sodium lauryl sulfates), and cationic (cetyl pyridinium chloride). Those skilled in the art of formulating coating compositions will be able to select that particular plasticizer most suitable in any particular application. It will be appreciated, as earlier suggested, that use of such a component in any specific case is entirely optional and will depend upon the desired flexibility in the cured coating. For example, it may be desirable to include a plasticizer in a coating composition containing an epoxy acrylate resin, as such compositions, in general, will be found, on curing, to be relatively rigid.

Leveling Agents: Examples of various leveling agents that can be used in the invention are: sucrose benzoate, copolymer 2-ethylhexyl acrylate and ethyl acrylate, calcium stearate, and nonionic surface active agents.

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Thixotropic Agents: Examples of various thixotropic agents that can be used in the invention are: aluminum, calcium and zinc salts of fatty acids such as lauric or stearic acid; fumed silica, hydrated siliceous material, calcium carbonate, magnesium oxide, high molecular weight polyacrylic acids, stearates, polysubstituted sorbitols, bentonite, and chrysotile asbestos. In general, such an additive will be found desirable in compositions used on radial leads, or in applications involving roll coating and dipping. Of the above, fumed silica will be found particularly satisfactory. Also, in addition to the above, it will often be found that any of the fillers above mentioned, will produce the desired thixotropic properties.

Flame Retardants: Examples of various flame retardants that can be used in the invention are: tetrabromo disphenol A-diacrylate, methyl pentachlorostearate, bis(tribromo-phenoloxy) ethane, aluminum trihydrate, antimony oxide, chlorinated paraffins, chlorinated cycloaliphatics, aromatic bromine compounds, phosphates, zinc borates, barium metaborates, ammonium floroborates, decabromodiphenyl oxide, ammonium bromide, and phosphorium bromide.

Adhesion Promoters: Examples of various adhesion promoters that can be used in the invention are: dimethylaminoethyl acrylate and methacrylate, diethlaminoethyl acrylate and methacrylate, silanes, titanates, allyl ether of cyclic ureido, zircoaluminate, lignocellulosic, and thiodigylcol

diacrylates and methacrylates. In some instances, where greater adhesion is required with a particular substrate coated, the adhesion will be obtained by first heating the substrate, and then applying the coating composition.

Stabilizers: Examples of various stabilizers that can be used in the invention are: 2-hydroxy-4-alkoxy benzophenones, 2(2 hydroxy phenyl) benzotriazole, salicylates, resorcinol monobenzoates, aryl or alkyl hydroxy benzoates, substituted oxanilides, substituted formamidine, 2,2 thiobis (4-octylphenolato)-n-butylamine nickel II, nickel salts of thiocarbamate, alkylated phenol phosphonate nickel, and tetramethyl pyperidine. Such compounds will be used as necessary to increase the pot life of the coating or ink composition.

Inhibitors: Examples of various inhibitors that can be used in the invention are: hydroquinone, p-methoxyphenol, alkyl and aryl-substituted hydroquinones and quinones, tert-butyl cathechol, pyrogallol, octadecyl-B-(hydroxy-3,5-di-t-butyl phenyl)-propionate, 2,6-di-tert,-butyl-4-methyl phenol, phosphorous acid, beta-napthol, 2,6-di-tert-butyl p-cresol, 2,2-methylene bis(4-ethyl-6-t-butylphenol), p-toluquinone, aryl and alkyl phosphites, and benzotriazoles. Addition of such components to the compositions of this invention will increase the shelf life, as desired.

Those skilled in the art of formulating coating compositions can readily determine the amounts of such optional additives, to be added to any particular composition of our invention, for any particular application and desired property. Such materials are, in general, present in the compositions in very minor concentrations, ranging from less than 5% by weight of the polymer composition, and usually less than 3% by weight of the polymer compositions. In any event, such additives are not included in any significant amount whereby the additives do not interfere with the cross-linking of the polymeric material or to lessen the good properties desired, and achieved, in the cured coating.

In preparing the coating compositions of this invention, the components are mixed together by conventional procedures used in the production of inks, and coating compositions. These procedures are so well known to those skilled in the art that they do not require further discussion here. However, it should be noted that when the composition is to be UV cured it will be necessary that the photoinitiator be incorporated into the coating compositions and that the curable coating formulation be mixed or blended under "safe light" such as a yellow light source to obviate or minimize photopolymerization.

The components can be mixed together and formulated into the desired composition using any conventional mixing apparatus, e.g. a Banbury or

Werner & Pfliderer mixer. The components can be added together in any sequence desired. Nevertheless, it is preferred that the surface active agent be added first to the prepolymer, monomer mixture followed by addition of the filler. Any processing apparatus used should obtain thorough mixing of the essential components and provide that the surface active agent is intimately and thoroughly dispersed throughout the polymer/monomer mixture.

In the more preferred manner of formulating the compositions of this invention, the filler will be pre-treated with a surface active agent. This can be accomplished by using filler coated in a previous operation, or by adding the surface active agent first to the mixer and next dispersing the filler therein.

The curable coating compositions of this invention can be applied to a suitable surface or substrate, e.g. an electrical and electronic component, by conventional means such as roll or dip coating, end pouring, spraying, etc. Curing, or photopolymerization of the coating compositions occurs on exposure of the compositions to any source of radiation emitting actinic radiation at a wavelength within the appropriate ultraviolet or visible spectral regions. Suitable sources of radiation include mercury, xenon, carbon arc and tungsten filament lamps, sunlight, etc. Exposures may be from less than about 4 seconds to 5 minutes or more depending upon the amounts of particular

polymerizable materials and photoinitiators being utilized, and depending upon the radiation source and distance from the source, and the thickness of the coating to be cured. The compositions may also be polymerized by exposure to electron beam irradiation without the need of photoinitiator or synergist in the composition. Generally speaking the dosage necessary is from less than 1 megarad to 100 megarad or more.

The thermoplastic coating compositions of this invention can be applied to a suitable surface or substrate as described above. The solvent can be evaporated from the system at ambient temperature or with the application of some heat.

The following examples, in which all parts are expressed by weight percent of total composition will serve to illustrate the invention more fully.

Example 1

UV Curable, Highly Thixothropic, Flame Retardant Conformal Coating for Electrical and Electronic Components

This example illustrates the effectiveness of the composition in coating pear-shaped electrical and electronic components such as tantalum capacitors.

The composition is prepared by mixing together the prepolymer, monomer, photoinitiators, sensitizers, wetting agent and dispersant in the weight percentages given below. The mixture is

preferably heated to 60°C whereby any solid photoinitiators are dissolved. When the photoinitiator is completely dissolved, the filler, flame retardant additives and pigment are added and mixing continued for 15 minutes while maintaining a temperature within the range of 60-80°C. The mixture is allowed to cool to ambient conditions before adding the peroxide and bubble breaker. Mixing is resumed at low shear and ambient temperatures until peroxide and bubble breaker are thoroughly mixed.

These compositions are coated onto dry slug tantalum capacitors by dipping the preheated capacitors at 85°C into the resin composition and withdrawing the units on chipboards with mechanical agitation. Units are then exposed to actinic radiation within the wavelength region of 185-400 nm for 5 seconds. The same units are subsequently dipped a second time and exposed again to the said radiation for 5 seconds. Coating thicknesses of 250 mils were necessary to ensure complete coverage of the "hockey stick" leads that make up the cathode and anode functions of the capacitor.

As the coating thickness of this electrical and electronic component is 250 mils, the coating is further subjected to a postheat treatment for 15 minutes at 85°C. This thermal treatment hastens the further cross-linking of the polymeric composition. Nevertheless, where the coating thickness is less than about 10 mils, no such thermal treatment is

needed, as the crosslinking of the polymeric composition is essentially completed during the UV curing.

The cured coating composition was evaluated according to usual techniques and was found to exhibit low shrinkage, high tensile and compression strength, as well as broad operating temperatures (-65 to 125°C) over 2000 hours of service life. In addition, the coating is able to withstand high temperature concomitant with high humidity conditions without seriously degrading the electrical behavior of the component. Table 7 illustrates the average electrical properties for twenty units of each of the compositions A-F set forth in Table 6 tested under high temperature (85°C) concomitant with high humidity (95%RH) conditions for 1,000 hours. In the table, electrical properties are better for the treated filler with dispersant added (composition A) and for the treated filler without dispersant (compositions B and C) than for the untreated filler (composition D). The untreated filler composition showed severe cracking after 500 hours at 125°C.

As this example shows, the compositions of this invention combine ease in handling with desired physical, thermal, electrical insulation and moisture resistance properties.

TABLE 6

Dry Slung Tantalum Capacitor Compositions

Composition (%wt.)

Materials	A	B	C	D	E	F
Prepolymer 1573	5.5	4.0	5.0	9.0		
Monomer DCPA	6.0	4.0	7.6	8.3		
TMPTA	6.5	12.5	10.7	8.0		
HDODA						
BM-605					1.5	1.5
HPA					5	5
PETA					6	6
Wetting Agent Fc430	0.8	1.3	1.3	0.8		
Photoinitiator & Sensitizers IR-651	2.0	2.1	2.1	2.1	1.5	1.5
ITX	1.0				0.7	0.7
EBP	1.5					
Dispersant RE-610	1.3				1.3	1.0
CC-42						0.3

TABLE 6 (CONT.)

Dry Slung Tantalum Capacitor Compositions

Materials	Composition (%wt.) A	B	C	D	E	F
Flame Retardants						
Oncor	2	2	2	2.0	1.8	1.8
DBDPO	5.7	5.7	5.7	5.7	5.4	5.4
Fillers						
*400-NYAD-10024 (Wallastonite)	66	62.8	61.5		75.8	65.8
*400-NYAD (Wallastonite)				61.5		
Monarch 1000						10.0
Pigment						
Dye	0.5	0.5	0.5	0.5	0.5	0.5
Peroxide						
TBP	5.0	3.5	2.0			
L-231	1				0.5	0.5
Bubble Breaker						
Air Out	0.1	0.1	0.1	0.1		

*400-NYAD-10024 is calcium metasilicate treated with 1%, Z-6030 silane (Dow Corning) at a 1% by weight silane per calcium metasilicate (Wallastokup)

Table 7

Electrical Properties

Humidity at 85°C, 95%RH, for 1,000 hours

Testing Done at 0.5V, 12Hz

Composition	Initial Electrical Values			Final Electrical Values		
	CAP(uf)	DF(%)	ESR(ohm)	CAP(uf)	DF(%)	ESR(ohm)
A	48.40	3.60	0.99	49.52	3.80	1.04
B	49.11	3.72	1.01	51.00	4.29	1.12
C	48.87	3.37	.91	50.70	3.89	1.02
D*	49.21	3.42	.92	51.57	6.74	1.66
E	48.6	3.61	.97	49.71	3.81	1.02
F	49.0	3.47	.93	52.0	5.63	1.46

*Test done for only 500 hours. Parts are severely split.

CAP - Capacitance in microfarads

DF - dissipation factor in percent

ESR - electrical series resistance in ohms

Example 2

Low Viscosity, Flame Retardant, UV Curable End Pour System for Smaller Electrical and Electronic Components

The compositions of this example are particularly suitable as protective coatings for smaller electrical and electronic components such as axial leaded film foil capacitors, hybrid circuits, printed circuit boards, transformers, delay lines, electrolytic capacitors, axial leaded resistors, etc., in which the composition is poured into the end of the tape wrapped unit, preformed casing, metal casing, etc.

The units, end poured in this example, are axial leaded film foil cylindrical capacitors whose diameter ranges from 0.0625 to 0.25 inches, and whose length ranges from 0.50 to 1.50 inches.

TABLE 8

End Pour Encapsulant

Composition (%wt.)

Materials	A	B	C	D	E	F	G	H
Prepolymer								
1573	10	9	10	9	10	9		
Monomer								
DCPA	12	20.3	13	21.1	12	20.3		
HDODA	14		14		14			
TMPTA		10.5		10.5		10.5		
HPA							7	7
PETA							7	7
Photoinitiator								
IR-651	2.1	2.1	2.1	2.1	2.1	2.1	1.5	1.5
Dispersant								
RE-610	1.0	0.9			1.0	1.0	1.2	1.0
CC-44								0.3
Pigment								
Dye	1.2	1.2	1.2	1.2	1.2	1.2	0.7	0.7
Flame Retardent								
Oncor	2	2	2	2	2	2	1.7	1.7
DBDPO	5.7	5.7	5.7	5.7	5.7	5.7	5.7	5.7
Filler								
NYAD-325	50	46.3						
NYAD-325-10022			50	46.3	50	46.3	74.5	64.4
Monarch 1000								10.0
Peroxide								
TBP	2	2	2	2	2	2	0.7	.07
Viscosity	550	1000	750	1200	400	800	10,000	15,000

(cps; centipoise)

Table 9
Electrical Properties
End Pour, "Mylar" Tape Wrapped Capacitors
Humidity at 75°C, 95%RH, for 100 hours

Composition	Initial Electrical Values			Final Electrical Values		
	CAP(uf)	DF(%)	ESR(ohm)	CAP(uf)	DF(%)	ESR(ohm)
A	10.41	.004	.01	10.46	.004	.01
B	10.37	.004	.01	10.38	.004	.01
C	10.30	.003	.01	10.28	.003	.01
D	10.39	.004	.01	10.41	.004	.01
E	10.37	.004	.01	10.41	.004	.01
F	10.21	.003	.01	10.28	.003	.01
G	10.38	.003	.01	10.41	.003	.01
H	10.31	.003	.01	10.34	.003	.01

The above compositions (A-H in Table 8) are generally applied by automatic, premeasured, volumetric dispensers. Other methods such as spray and dip application before potting are also possible. Coating thicknesses generally range from 0.010 to 0.500 inches. The coating is subjected to UV curing, as before disclosed in Example 1, by exposure for 6 seconds under a medium pressure mercury vapor lamp whose wavelength for curing ranges from 185-400 nm. Afterwards, the coating was subjected to a post heat treatment of 85°C for 15 minutes, to effect complete cure.

Table 9 illustrates the electrical properties taken before and after humidity (95% RH) testing at 75°C for 100 hours. The data is an average of twenty axial leaded film foil capacitor values. Compositions A through H gave good electrical values. These compositions are particularly effective in eliminating bubbles and pin-holes often created by entrapped air in the end-pour units.

Example 3

Low Viscosity, UV Curable
Potting System

The compositions in this example are highly suitable for use in applications involving potting and coating the same electrical components as in Example 2, e.g. axial leaded film foil capacitors, hybrid circuits, printed circuit boards, electrolytic capacitors, transformers, delay lines, resistors,

etc. The compositions eliminate, or at least lessen the formation of, pin-holes and bubbles that often form in potting applications due to outgassing of entrapped air during the potting application. The resin system cures quickly forming an air tight seal, hence, preventing escape of air within the electrical and electronic package. Humidity resistance is a characteristic property for the composition of this example as well as its adhesion to aluminum, copper, gold, palladium, and silver.

The compositions A-H set forth in Table 10 are applied as in Example 2. Units that are end poured in this Example are axial leaded film foil cylindrical capacitors whose diameter ranged from 1.25 to 2.0 inches.

Table 10

End Pour Encapsulant

Composition (%wt.)

Materials	A	B	C	D	E	F	G	H
Prepolymer								
1573	10	9	10	9	10	9		
Monomer								
DCPA	12	20.3	12	20.3	12	20.3		
HDODA	14		15		14			
TMPTA		10.5		11.4		10.5		
HPA							5	5
PETA							7	7
Photoinitiator								
IR-651	2.1	2.1	2.1	2.1	2.1	2.1	1.5	1.5
Dispersant								
RE-610	1.0	0.9			1.0	0.9	1.3	1.2
CC-42								0.2
Filler								
NYAD-325	58.9	55.2					84.5	74.4
NYAD-325-10022			58.9	55.2	58.9	55.2		
Monarch 1000								10.0
Peroxide								
TBP	2	2	2	2	2	2	0.7	0.7
Viscosity(cps)	1700	2000	2300	2500	1500	1800	9000	14,000

Table 11

Electrical Properties

End Pour, "Mylar" Tape Wrapped Capacitors

Humidity at 75°C, 95%RH, for 100 hours

Composition	Initial Electrical Values			Final Electrical Values		
	CAP(uf)	DF(%)	ESR(ohm)	CAP(uf)	DF(%)	ESR(ohm)
A	9.87	.05	.01	9.94	.05	.01
B	10.21	.04	.01	10.18	.04	.01
C	9.92	.03	.01	9.93	.03	.01
D	9.85	.04	.01	9.97	.04	.01
E	9.92	.04	.01	9.98	.04	.01
F	9.84	.03	.01	9.96	.03	.01
G	9.96	.04	.01	9.99	.04	.01
H	9.89	.04	.01	9.98	.04	.01

The resin compositions A-H of this example polymerize to a hard thermoset plastic within 6 seconds of exposure to actinic radiation within the wavelength range of 185-400 nm, from a mercury vapor lamp as earlier disclosed. A postheat treatment for 15 minutes at 85°C was necessary to effect complete cure.

The coated film foil capacitor was subjected to operating temperatures between -55 to 105°C for 1000 hours without delamination of the coating from the metallized surface. The pigmentation of the resin did not affect the cure properties of the coating, and did not significantly decrease cure speed.

Table 11 illustrates the electrical values for the said capacitor units after humidity (95% RH) testing at 75°C for 100 hours. Measurements are an average of twenty film foil capacitor values. Compositions A through H gave good electrical values.

Example 4

Low Viscosity, UV Curable Coating System for Electrical and Electronic Components

The compositions of this example (compositions A-F set forth in Table 12) are useful for potting or casting electrical and electronic components and may be used in combination with the compositions of Example 3. The rapid cure of the composition in Example 3 allows application of this

coating to a substrate that is free of bubbles and pin-holes, often generated during the potting process due to entrapped air within the electrical and electronic components.

The compositions of Example 4 are applied in a manner as described in Example 2. The end pour units of this example are cylindrical film foil axial leaded capacitors, with a diameter whose range is from 1.0 to 2.50 inches and a length whose range is from 1.25 to 2.0 inches.

Table 12

End Pour Encapsulant

Composition (%wt.)

Materials	A	B	C	D	E	F
Prepolymer						
1573		10		10		10
3600	9		9		9	
Monomer						
DCPA	10.2	4	11.2	5.0	10.2	4
TMPTA	10.5	15	10.5	15	10.5	15
Photoinitiator						
IR-651	2.1	2.1	2.1	2.1	2.1	2.1
Dispersant						
RE-610	1.0			1.0	1.0	
Pigment						
Dye	1.2	1.2	1.2	1.2	1.2	1.2
Flame Retardant						
Oncor	2	2	2	2	2	2
DBDPO	5.7	5.7	5.7	5.7	5.7	5.7
Filler						
NYAD-325	56.3	57				
NYAD-325-10022			56.3	57	56.3	57
Peroxide						
TBP	2	2	2	2	2	2
Viscosity(cps)	9100	13,200	10,000	14,100	8000	11,100

Table 13

Electrical Properties

End Pour, "Mylar" Tape Wrapped Capacitors

Humidity at 75°C, 95%RH, for 100 hours

Composition	Initial Electrical Values			Final Electrical Values		
	CAP(uf)	DF(%)	ESR(ohm)	CAP(uf)	DF(%)	ESR(ohm)
A	9.42	.05	.01	9.63	.05	.01
B	9.32	.05	.01	9.52	.05	.01
C	8.22	.04	.01	8.32	.04	.01
D	8.18	.06	.01	8.26	.06	.01
E	8.20	.07	.01	8.29	.07	.01
F	9.61	.05	.01	9.73	.05	.01

These compositions cure within 6 seconds exposure to radiation from a mercury vapor lamp as disclosed in Example 1. A postheat treatment for 15 minutes at 85°C is required to effect complete cure.

The said capacitors of Example 4 are humidity (95% RH) tested at 75°C for 100 hours. The data illustrated in Table 13, is an average of twenty film foil capacitor values. Compositions A through F gave good electrical values for coating thicknesses that ranged from 0.010 to 0.090 inches.

The cured coatings exhibit uniform grafting to the polymer coating of Example 3, and good adhesion to metallic substrates.

In many cases, a satisfactory coating may be achieved without pre-coating the electrical and electronic components with the composition of Example 3.

Example 5

Flame Retardant, UV Curable Conformal Coatings for Hybrid Circuits, Radial Film Foil Capacitors, and Ceramic Capacitors

The compositions of this example (compositions A-F set forth in Table 14) are useful in coating hybrid circuits, radial film foil capacitors and ceramic capacitors due to their ability to withstand high humidity environments and temperature extremes from -55 to 125°C.

Table 14

Hybrid Circuit, Radial Film Foil Capacitor,

Ceramic Capacitor Encapsulant

Composition (%wt.)

Materials	A	B	C	D	E	F
Prepolymer						
1537	8.5	6.3	7.6			
Monomer						
DCPA	7.5	4.0	4.0			
TMPTA	4.3	4.7	4.7			
BM-605				2.0		
HPA				5.0	5.0	5.0
PETA				6.0	6.0	6.0
Wetting Agent						
Fc-430	2	0.8	0.8			
Plasticizer						
L-722	3.5	3.0	3.0			
Photoinitiator						
IR-651	2.8	2.0	2.0	1.5	1.5	1.5
ITX		2.0	2.0	1.0		
MK			0.7			
EPB		2.0				
Dispersant						
RE-610		1.6	1.6	1.4	1.3	1.4
CC-42					0.2	
Pigment						
Dye	0.8	0.6	0.6	0.5		0.5

Table 14 (continued)

Materials	A	B	C	D	E	F
Flame Retardant						
Oncor	2	1.9	1.9	1.8	1.8	
DBDPO	10.5	5.7	5.7	5.7	5.7	
Filler						
400-NYAD-10024	52.6	64.3	64.3	74.3	67.8	84.9
Monarch 1000					10	
Bubble breaker						
Air Out		0.1	0.1	0.1		
Peroxide						
TBP	5.5					
L231		1.0	1.0	0.7	0.7	0.7

Table 15
Electrical Properties
Ceramic Capacitors
Humidity at 85°C, 95%RH, for 1000 hours

Composition	Initial Electrical Values			Final Electrical Values		
	CAP(uf)	DF(%)	ESR(ohm)	CAP(uf)	DF(%)	ESR(ohm)
A	48.12	1.21	0.40	47.79	1.21	0.39
B	48.95	1.19	0.39	49.12	1.17	0.42
C	49.14	1.29	0.42	49.46	1.32	0.44
D	49.12	1.30	0.44	49.40	1.31	0.44
E	48.66	1.19	0.40	49.12	1.22	0.42
F	48.67	1.21	0.41	49.23	1.26	0.44

The electrical and electronic components, e.g., hybrid circuits, radial film foil capacitors, and ceramic capacitors, are dip-coated into the resin composition of Example 5, and curing is accomplished by exposing the electrical and electronic units to actinic radiation as described in Example 1, for 6 seconds. A second coating application is necessary to ensure complete electrical and electronic component coverage. After a second costing, the units are exposed to actinic radiation as previously described. The radiation cure is followed by a heat treatment in a convection oven at 85°C for 15 minutes. The amount of resin used depends upon the configuration and size of the electrical and electronic component. Also, the resin thickness applied in each case depends upon the type of components surface mounted on the hybrid circuit, upon the type of attachment of lead wires to the ceramic capacitor and size of the ceramic capacitor, and upon the size, and shape, and roughness of the radial film foil capacitor. Coating thicknesses vary anywhere from 0.010 to 0.225 inches per dipping application.

Electrical values for disc shaped ceramic capacitors of Example 5 are illustrated in Table 15. These measurements are an average of twenty ceramic capacitor values. Compositions A through F gave good electrical values after humidity (95% RH) testing at 85°C for 1000 hours.

Example 6

UV Curable Printing Ink for
Heat Sensitive Substrate

An ink composition is prepared by combining the following Table 16 components in the amounts indicated in Table 16.

The viscosity of this composition is determined, in accordance with ASTM D115-82, to be 90,000 cps to 300,000 at 25°C.

The composition is printed onto polypropylene, mylar, and polystyrene (heat sensitive), in accordance with usual techniques, after which it is exposed to actinic radiation as previously described in Example 1, for 2 seconds.

These ink formulations are tested according to Military Standards 883C and all inks pass.

Table 16

Ink Formulations for Heat Sensitive Substrates

Composition (% wt.)

Material	A	B	C	D	E	F	G	H	I
Prepolymer									
1570	50	50	50	50	50	40			
Dispersant									
Re-610	0.04	0.04	0.04	0.04	0.04	0.04	1.6		0.5
CC-42								1.4	0.8
Monomer									
TMPTA	32.96	32.96	32.96	32.96	32.96	32.6			
HPA							5	5	5
PETA							6	6	6
Photoinitiator									
IR-651		6	6	6	6	6			
Lucirin LR8728						0.5	0.5		
ITX		2.5	2.5	2.5	2.5	2.5		1.5	1.5
BP						1.5	1.5	1.5	1.5
DMAEA	3.5	3.5	3.5	3.5	3.5	2.0	2.0	2.0	2.0

Table 16 (continued)

Material	A	B	C	D	E	F	G	H	I
Thixotropic agent									
Cab-o-sil	3	3	3	3	3	3.0	1.0	1.0	
Pigment									
Black F2302	2							81.6	52.7
Blue F5203		2							
Green			2						
Brown				2					
Yellow					2				
TiO_2						10	30		30
ZnS						10	52.4		

Table 17

Ink Formulations for Non-Heat Sensitive Substrates

Composition (% wt.)

Material	A	B	C	D	E	F	G	H
Prepolymer								
1570	45	45	45	45	45			
Dispersant								
RE-610	0.1	0.1	0.1	0.1	0.1	1.6		0.5
CC-42							1.4	0.8
Monomer								
TMPTA	34.9	34.9	34.9	34.9	34.9			
HPA						4	4	5
PETA						6	5	6
Photoinitiator								
IR-651	6	6	6	6	6			
Lucirin LR8728						0.5		
ITX	2.5	2.5	2.5	2.5	2.5		1.0	1.0
BP						1.0	1.0	1.0
Thixotropic agent								
Cab-o-sil	3	3	3	3	3	1.0	1.0	
Pigment								
Black	5						84.6	53.7
Blue		5						
Green			5					
Brown				5				
Yellow					5			
T_iO_2						30		30
ZnS						53.9		

Example 7

UV Curable Composition for
Non-Heat Sensitive Substrate

A further ink composition is prepared for non-heat sensitive substrates by mixing together the following Table 17 components illustrated in Table 17.

These compositions, which had viscosity of 90,000 cps at 25°C, are printed onto an aluminum can and cured as in the previous example for 8 seconds. The ink compositions exhibit properties comparable to those of the previous example.

Example 8

Thermoplastic polymer containing compositions were prepared as shown in Tables 18-20. The thermoplastic polymer used is Butvar (B79) as described in Table 18 and the volume % filler ranged from 46.1 to 95%.

Table 18

NYAD and Carbon Fillers

Experiment Number

Materials	1 %wt.	2 %wt.	3 %wt.	4 %wt.	5 %wt.	6 %wt.	7 %wt.
Polymer							
Butvar B79	7.0	7.0	7.0	7.0	7.0	7.0	7.0
Solvents							
MEK.	12.0	12.0	12.0	12.0	12.0	12.0	12.0
Tol.	18.0	18.0	18.0	18.0	18.0	18.0	18.0
DBP	1.3	1.3	1.3	1.3	1.3	1.3	1.3
Fillers							
NYAD 400	61.1	51.15	41.2	31.1	21.2	11.2	
CB(1000)		10.0	20.0	30.15	40.1	50.2	61.4
Dispersants							
Re-610	0.6	0.5	0.4	0.3	0.2	0.1	
CC-42		0.05	0.1	0.15	0.2	0.2	0.3
VOLUME % FILLER	69.2	71.2	72.9	74.5	75.9	77.3	78.4

Table 19

NYAD and Copper Fillers

Experiment Number

Materials	1 %wt.	2 %wt.	3 %wt.	4 %wt.	5 %wt.	6 %wt.
<u>Polymer</u>						
Butvar B79	7.0	7.0	7.0	7.0	7.0	7.0
<u>Solvents</u>						
MEK.	12.0	12.0	12.0	12.0	12.0	12.0
Tol.	18.0	18.0	18.0	18.0	18.0	18.0
DBP	1.3	1.3	1.3	1.3	1.3	1.3
<u>Fillers</u>						
NYAD 400	51.15	41.2	31.1	21.2	11.2	
Copper R290	10.0	20.0	30.15	40.1	50.2	61.4
<u>Dispersants</u>						
Re-610	0.5	0.4	0.3	0.2	0.1	
CC-42	0.05	0.1	0.15	0.2	0.2	0.3
VOLUME % FILLER	67.0	64.3	61.2	57.5	53.0	46.1

Table 20

NYAD and Nickel Fillers

Experiment Number

Materials	1 %wt.	2 %wt.	3 %wt.	4 %wt.	5 %wt.	6 %wt.
Polymer						
Butvar B79	7.0	7.0	7.0	7.0	7.0	7.0
Solvents						
MEK.	12.0	12.0	12.0	12.0	12.0	12.0
Tol.	18.0	18.0	18.0	18.0	18.0	18.0
DBP	1.3	1.3	1.3	1.3	1.3	1.3
Fillers						
NYAD 400	51.15	41.2	31.1	21.2	11.2	
Nickel PN200	10.0	20.0	30.15	40.1	50.2	61.4
Dispersants						
Re-610	0.5	0.4	0.3	0.2	0.1	
CC-42	0.05	0.1	0.15	0.2	0.2	0.3
VOLUME % FILLER	95.0	94.6	94.2	93.6	93.3	91.3

It will now be apparent to those skilled in the art that other embodiments, improvements, details, and uses can be made consistent with the letter and spirit of the foregoing disclosure and within the scope of this patent, which is limited only by the following claims, construed in accordance with the patent law, including the doctrine of equivalents.

What is claimed is:

1. A highly, filled composition, comprising:

a polymeric material;

at least one filler, and

at least one surface active agent;

wherein one of the surface active agent, or filler has an overall pH greater than or equal to 7 and the other of the surface active agent or filler has an overall pH less than or equal to 7, provided that both overall pH's do not equal 7, the pH relationship between the surface active agent and filler enhancing the filler loading of the composition.

2. The highly, filled composition of claim 1 wherein the polymeric material includes a thermoset polymer.

3. The highly, filled composition of claim 2 wherein the composition further comprises a photoinitiator.

4. The highly, filled composition of claim 2 wherein the polymeric material includes a prepolymer.

5. The highly, filled composition of claim 4 wherein the prepolymer is an aromatic epoxy acrylate.

6. The highly, filled composition of claim 5 wherein the aromatic epoxy acrylate is a diacrylate ester of bisphenol A epoxy.

7. The highly, filled composition of claim 4 wherein the polymeric material includes a monomer.

8. The highly, filled composition of claim 7 wherein the monomer is selected from the group consisting of trimethylol propane triacrylate, 1, 6 hexanediol diacrylate, dicyclopentenylacrylate, hydroxypropyl acrylate, and dimethylaminoethyl acrylate.

9. The highly, filled composition of claim 3 wherein the photoinitiator is selected from the group consisting of 2-isopropyl-thioxanthone, 2-2 dimethoxy-2, phenylacetophenone, 2-hydroxy-2 methyl-1-phenyl propan-1-one, 2-ethylhexyl p-(N,N-dimethylamino) benzoate, and 2-(dimethyl amino) ethyl benzoate.

10. The highly, filled composition of claim 1 wherein the polymeric material includes a thermoplastic polymer.

11. The highly, filled composition of claim 10 wherein the composition further comprises a solvent.

12. The highly, filled composition of claim 11 wherein the thermoplastic polymer is selected from the group consisting of polyvinylbutryate and n-butylpolymethacrylate.

13. The highly, filled composition of claim 11 or 12 wherein the solvent is selected from the group consisting of methylethylketone, toluene, isopropyl alcohol and xylene.

14. The highly, filled composition of claim 1, 2 or 10 wherein the filler is selected from the group consisting of calcium metasilicate, titanium dioxide and zinc sulfide.

15. The highly, filled composition of claim 1, 2 or 10 wherein the composition contains two surface active agents.

16. The highly, filled composition of claim 1, 2, 10 or 11 wherein the composition contains two fillers.

17. The highly, filled composition of claim 1, 2 or 11 wherein one of the surface active agents is an organic phosphate ester.

18. The highly, filled composition of claim 1, 2 or 10 wherein the filler used has been precoated with a surface active agent.

19. The highly, filled composition of claim 18 wherein the precoated filler is calcium metasilicate and the surface active agent is silane.

20. The highly, filled composition of claim 19 wherein the silane is methacryloxy propyltrimethoxy-silane.

21. The highly, filled composition of claim 13 wherein the organic phosphate acid ester is selected from the group of nonyl nonoxynol-7-phosphate, nonyl nonoxynol-10-phosphate, nonyl nonoxynol-15-phosphate,

nonoxynol-9-phosphate, nonoxynol-4-phosphate, trideceth-6-phosphate, trideceth-4-phosphate, trideceth-7-phosphate, polyoxyethylene lauryl ether phosphate, polyoxyethylene phenyl ether phosphate, isoamyl alcohol ethoxylated phosphate, polyoxyethylene butoxyethyl ether phosphate, polyoxyethylene hexyl ether phosphate, oleic acid, palmitic acid and stearic acid.

22. The highly, filled composition of claim 1, 2 or 10 wherein the filler is carbon black.

23. The highly, filled composition of claim 1, 2 or 10 wherein the filler is an alloy of chromium/copper/manganese/molybelenum, or cobalt/chromium/aluminum/or chromium/cobalt/-magnesium/aluminum or chromium/iron/silicon, or mickel/tin/titanium.

24. The highly, filled composition of claim 1, 2 or 10 wherein the filler is selected from the group consisting of copper, aluminum, iron oxide, silver, gold, palladium, platinum, iridium, tin, lead, cobalt, iron, magnesium, silicon, nickel, manganese, chromium, tungsten and titanium.

25. The highly, filled composition of claim 1, 2 or 10 wherein the surface active agent is a quaternary ammonium salt.

26. The highly, filled composition of claim 1, 2 or 10 wherein the filler is calcium metasilicate and the surface active agent is silane.

27. The highly, filled composition of claim 1, 2 or 10 wherein the filler is calcium metasilicate and the surface active agent is an organic phosphate ester.

28. The highly, filled composition of claim 1 or 10 wherein the filler is calcium metasilicate and the surface active agent is an organic acid and/or alcohol.

29. The highly, filled composition of claim 1, 2 or 10 wherein the filler is carbon black and the surface active agent is an quaternary ammonium salt, alcohol or combination thereof.

30. The highly, filled composition of claim 1, 2 or 10 wherein the filler is a metal alloy and the surface active agent is a quaternary ammonium salt, alcohol, or combinations thereof.

31. The highly, filled composition of claim 1, 2 or 10 wherein the composition further comprises a flame retardant component.

32. The highly, filled composition of claim 1, 2 or 10 wherein the composition further comprises an inhibitor component.

33. The highly, filled composition of claim 1, 2 or 10 wherein the composition further comprises a thixotropic agent.

34. A highly, filled composition, comprising:
a polymeric material;
at least one filler, and
at least one surface active agent;
wherein the volume % of filler in the composition is sufficiently high to impart durability into a coating formed from the composition.

35. The highly, filled composition of claim 34 wherein the polymeric material includes a thermoset polymer.

36. The highly, filled composition of claim 34 wherein the polymeric material includes a thermoplastic polymer.

37. The highly, filled composition of claim 35 or 36 wherein the volume % filler is greater than about 50.

38. The highly, filled composition of claim 37 wherein the volume % filler is greater than about 60.

39. The highly, filled composition of claim 38 wherein the volume % of filler is greater than about 75.

40. The highly, filled composition of claim 39 wherein the volume % of filler is greater than about 90.

41. A method of preparing a highly, filled composition, comprising:

selecting a polymeric material;

selecting at least one filler having a first overall pH;

selecting at least one surface active agent having a second overall pH; and

mixing the polymeric material, filler(s) and surface active agent(s) forming a highly, filled composition, whereby one of the first and second overall pH's is greater than or equal to 7 and the other of the first and second overall pH's is less than or equal to 7 provided that both overall pH's do not equal 7.

42. The method of claim 41 wherein the polymeric material includes a thermoset polymer.

43. The method of claim 41 wherein the polymeric material includes a thermoplastic polymer.

44. The method of claim 42 or 43 wherein the filler has been pretreated with a surface active agent.

45. An electrical or electronic component coated with a composition as claimed in any one of claims 1, 2, 10 and 34.

46. A substrate painted with ink having a composition as claimed in any one of claims 1, 2, 10 or 34.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US92/06116

A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) :CO8K 3/00; CO8F 2/46
US CL :522/76, 77,79,81,83,103
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : NONE

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

APS, CHEMICAL ABSTRACTS VINYL, EPOXY ACRYLATE, PHOTOINITIATOR FILLER, SURFACE ACTIVE AGENT

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 4,459,193 (RATCLIFFE ET AL)10 JULY 1984, COL. 4, AND CLAIMS	1-46
Y	US, A, 4,407,984 (RATCLIFFE ET AL) 04 OCTOBER 1983, COL. 4 AND CLAIMS.	1-46
Y	US, A, 4,205,018 (NAGASAWA ET AL) 27 MAY 1980, COLS. 3-4.	1-46
Y	US, A, 4,052,280 (MCGINNIS) O4 OCTOBER 1977, COLS. 3-5.	1-46
Y,E	US, A, 5,134,175 (LUCEY) 28 JULY 1992, ENTIRE DOCUMENT.	1-46

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be part of particular relevance

"E" earlier document published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search 09 SEPTEMBER 1992	Date of mailing of the international search report [illegible] NOV 1992
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